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Efficient one-pot synthesis of 2,3-dihydroquinazoline-4(1*H*)-ones promoted by $FeCl_3$/neutral Al_2O_3

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Abstract

2,3-Dihydroquinazolin-4(1*H*)-one derivatives have been synthesized via one-pot reaction of isatoic anhydride, aromatic aldehyde, and ammonium acetate catalyzed by $FeCl_3$/neutral Al_2O_3 in *tert*-butanol under reflux conditions. Inexpensive and easily available reagents, convenient work-up procedure, reusable catalyst, and moderate to good yield are the salient features of this protocol.

Keywords 2,3-Dihydroquinazolin-4(1*H*)-one · $FeCl_3$/neutral Al_2O_3 · One-pot synthesis · Isatoic anhydride · Aldehydes

Introduction

2,3-Dihydroquinazolin-4(1*H*)-one derivatives are an important class of heterocycles containing nitrogen atom that exhibit a wide range of pharmacological and biological activities. Aquamox, evodiamine, fenquizone, metolazone, etc. are representative drugs containing quinazolinone core scaffold (Fig. 1). These quinazolinones can display a wide range of medicinal properties, including antibacterial [1], antiviral [2], antitumor [3], antihistamine [4], antiinflammatory [5–7], antiosteoporosis [8], anticonvulsant [9], antihypertensive [10], antidefibrillatory [11], and anticancer

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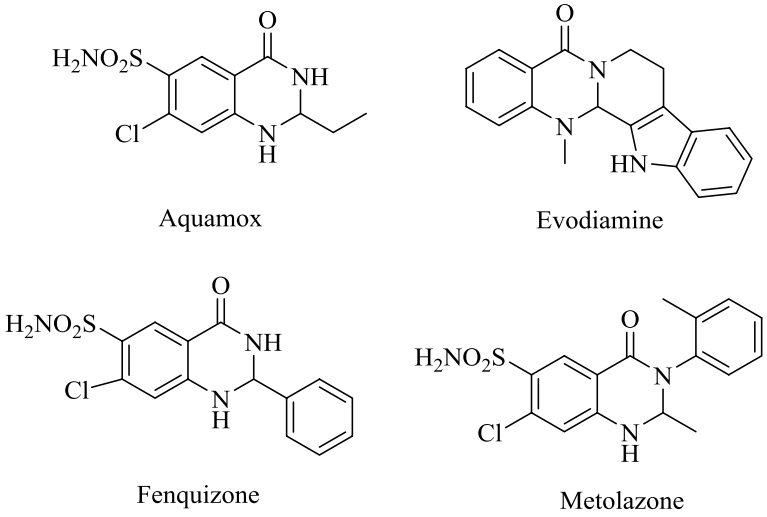


Fig. 1 Representative example quinazolinone drugs

effects [12] as well as potent human immunodeficiency virus (HIV)-1 reverse transcriptase inhibition [13, 14]. On the other hand, 2,3-dihydroquinazolin-4(1*H*)-one derivatives can be easily oxidized to 2-substituted-4(3*H*)-quinazolinones, which are important building blocks in synthesis of significant biologically active heterocyclic compounds. Hence, synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives is currently of great interest. In recent years, protocols for preparation of these compounds have been carried out in different ways [15]. Among them, the typical procedure includes condensation of anthranilamide with structurally diverse aldehydes or ketones in presence of various catalysts such as 2-morpholinoethanesulfonic acid [16], Co-carbon nanotubes (CNTs) [17], succinimide-*N*-sulfonic acid (SuSA) [18], propylphosphonic anhydride [19], poly(VPyPS)-PW [20], $HgCl_2$ [21], SiO_2-$H_3PW_{12}O_{40}$ [22], α-chymotrypsin [23], and nanocatalysts [24–28]. A more attractive and atom-efficient strategy for preparation of these compounds is one-pot, three-component reaction of isatoic anhydride, aldehydes, and ammonium acetate or primary amine. A number of catalysts, such as magnetic Fe_3O_4 nanoparticles [29], Al/Al_2O_3 nanoparticles [30], ethylenediamine diacetate [31], citric acid [32], silica-bonded *N*-propylsulfamic acid [33], silica-bonded *S*-sulfonic acid [34], $SrCl_2{\cdot}6H_2O$ [35], Cu-CNTs [36], H_3PO_4/Al_2O_3 [37], and dodecylbenzenesulfonic acid [38], have been utilized to accomplish this transformation. These synthetic strategies are useful to facilitate synthesis of the desired compounds in many ways. However, some of these methodologies are associated with different drawbacks such as long reaction time, tedious work-up, low yield, and toxic catalyst. Therefore, development of a mild, simple, efficient, and general method for synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones from readily available reagents remains one of the major challenges facing researchers in the field of organic synthesis.

Recently, development and utilization of supported catalysts have received increasing attention [39]. Such catalysts are efficient in organic synthesis, offering

the advantages of noncorrosivity, high selectivity, mild reaction conditions, and easy separation from the reaction system. As reported in previous papers, $FeCl_3/Al_2O_3$ has been applied in synthesis of 1,1′-binaphthalene-2,2′-diol [40] and diphenylmethane [41], esterification of *tert*-butanol [42], synthesis of 2-amino-2-hydroxy-1,1′-binaphthyl [43], desulfurization [44], and synthesis of 2-substituted benzimidazoles [45], affording the target products in higher yields.

In continuation of our research interest in synthesis of *N*–heterocyclic compounds with pharmacological activity using environmentally benign catalysts [45–49], we report herein a practical method for synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones via one-pot three-component condensation of isatoic anhydride, aldehydes, and ammonium acetate in presence of catalytic amount of $FeCl_3$/neutral Al_2O_3 using *tert*-butanol as solvent under reflux conditions (Scheme 1).

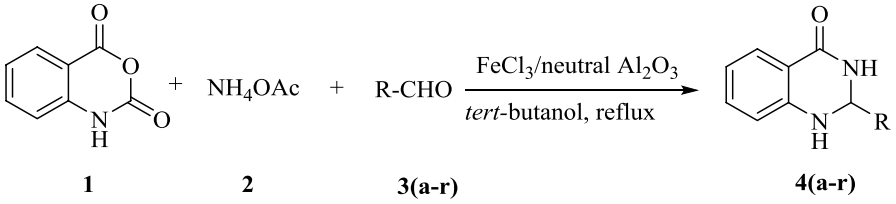
Experimental

Apparatus, materials, and measurements

All chemicals were commercially available and used without further purification. $FeCl_3$/neutral Al_2O_3 (10 % w/w) was prepared in accordance with a previously reported procedure [45]. All yields refer to isolated products after column chromatography on silica gel (200–300 mesh). Melting points were obtained using a TECH X-4 apparatus and are uncorrected. The reaction procedure was monitored in real time by thin-layer chromatography (TLC) plates visualized using ultraviolet light (254 nm). Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 600 (600 MHz for ^{1}H and 150 MHz for ^{13}C) instrument. Chemical shifts are expressed in ppm using tetramethylsilane (TMS) as internal standard with dimethylsulfoxide (DMSO)-d_6 as solvent; coupling constants (*J*) are reported in Hz. Mass-spectrometric data were collected on an Agilent Technologies 6310 ion trap LC/MS. Infrared (IR) spectra were measured on a Bruker Tensor-27 spectrophotometer using KBr pellets.

General procedure for synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

To a mixture of isatoic anhydride (2 mmol), aldehyde (2.4 mmol), and ammonium acetate (3.2 mmol) in *tert*-butanol (10 mL), $FeCl_3$/neutral Al_2O_3 (100 mg) was



Scheme 1 One-pot three-component synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

Table 1 Effect of different solvents on reaction of isatoic anhydride, benzaldehyde, and ammonium acetate

Entry	Solvent	Time (h)	Yield (%)[a]	TOF[b] (h^{-1})
1	Solvent-free	3	19	2.06
2	MeOH	3	26	2.82
3	*n*-Propanol	3	12	1.30
4	*n*-Butanol	3	17	1.84
5	EtOH	3	51	5.52
6	CH_2Cl_2	3	58	6.28
7	THF	3	58	6.28
8	$CHCl_3$	3	60	6.50
9	H_2O	3	62	6.72
10	1,4-Dioxane	3	72	7.80
11	DMF	3	69	7.47
12	Ethyl acetate	3	81	8.77
13	*tert*-Butanol	3	92	9.97

Experimental conditions: isatoic anhydride (2 mmol), benzaldehyde (2.4 mmol), ammonium acetate (3.2 mmol), and $FeCl_3$/neutral Al_2O_3 (100 mg) in 10 mL of different solvents under reflux

[a]Isolated yield based on isatoic anhydride

[b]Defined as moles of product per mole of catalyst per hour

DMF, dimethylformamide; THF, tetrahydrofuran; TOF, turnover frequency

Table 2 Effect of catalyst amount on reaction of isatoic anhydride, benzaldehyde, and ammonium acetate

Entry	$FeCl_3$/neutral Al_2O_3 (mg)	Time (h)	Yield (%)[a]
1	0	3	65
2	20	3	85
3	40	3	86
4	60	3	90
5	80	3	91
6	100	3	92
7	120	3	92

Experimental conditions: isatoic anhydride (2 mmol), benzaldehyde (2.4 mmol), and ammonium acetate (3.2 mmol) in 10 mL *tert*-butanol under reflux

[a]Isolated yield based on isatoic anhydride

added, then the reaction mixture was stirred at reflux temperature for appropriate time as indicated in Tables 1, 2, 3, and 4. After reaction completion as indicated by TLC (dichloromethane:ethyl acetate, 4:1), the catalyst was separated by filtration and washed with ethyl acetate. The solvent was evaporated under reduced pressure, and the products were further purified by chromatography eluted with ethyl acetate/dichloromethane (1:9) to afford 2,3-dihydroquinazolin-4(1*H*)-ones in good

Table 3 Effect of molar ratio on model reaction

Entry	Molar ratio[a]	Time (h)	Yield (%)[b]
1	1.0:1.0:1.6	3	87
2	1.0:1.0:1.8	3	73
3	1.0:1.1:1.6	3	89
4	1.0:1.1:1.8	3	90
5	1.0:1.2:1.0	3	79
6	1.0:1.2:1.2	3	83
7	1.0:1.2:1.4	3	88
8	1.0:1.2:1.6	3	92
9	1.0:1.2:1.8	3	92
10	1.0:1.3:1.4	3	85
11	1.0:1.3:1.6	3	85
12	1.0:1.3:1.8	3	92

[a]Isatoic anhydride/benzaldehyde/ammonium acetate(mmol/mmol/mmol)

[b]Isolated yield based on isatoic anhydride

Table 4 Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones in presence of $FeCl_3$/neutral Al_2O_3

Entry	R	Product	Time (h)	Isolated yield (%)[a]	Melting point (°C)	
					This study	Literature value
1	C_6H_5	**4a**	3	92	207–210	216–218 [17]
2	4-$NO_2C_6H_4$	**4b**	6	68	> 300	300–302 [30]
3	3-$NO_2C_6H_4$	**4c**	3	76	199–201	196–198 [17]
4	2-$NO_2C_6H_4$	**4d**	3	76	187–188	186–188 [22]
5	4-$CH_3C_6H_4$	**4e**	4	92	225–228	225–227 [17]
6	4-$MeOC_6H_4$	**4f**	5.5	89	192–194	193–195 [37]
7	4-ClC_6H_4	**4g**	3.5	88	200–203	205–206 [17]
8	3-ClC_6H_4	**4h**	4	89	188–190	185–187 [22]
9	2-ClC_6H_4	**4i**	4	87	204–206	202–204 [16]
10	4-OHC_6H_4	**4j**	7	54	272–274	275–277 [22]
11	3-OHC_6H_4	**4k**	5	38	202–205	206–207 [23]
12	4-FC_6H_4	**4l**	5	76	198–200	202–204 [18]
13	4-$Me_2NC_6H_4$	**4m**	3	90	214–216	209–212 [24]
14	2,4-$Cl_2C_6H_3$	**4n**	2	76	169–170	165–167 [35]
15	Furfural	**4o**	4	81	163–164	166–167 [16]
16	$C_6H_5CH{=}CH$	**4p**	5	79	163–166	198–200 [22]
17	3,4,5-$(CH_3O)_3C_6H_2$	**4q**	4	82	189–190	–
18	3,4-$Cl_2C_6H_3$	**4r**	3	90	197–198	–
19	2,4-$(NO_2)_2C_6H_3$	–	2	No product		
20	2,3,4-$(CH_3O)_3C_6H_2$	–	4.5	No product		
21	$CH_3(CH_2)_2$	–	4	No product		
22	$CH_3(CH_2)_5$	–	4	No product		

[a]Isolated yield based on isatoic anhydride

to excellent yield. The authenticity of the products was confirmed by IR, ^{1}H NMR, ^{13}C NMR, and MS and comparing their melting points with literature values.

Spectral and physical data for compounds

2-Phenyl-2,3-dihydroquinazolin-4(1*H*)-one (**4a**) was isolated as white crystals; m.p.: 207–210 °C; IR (KBr): 3302, 3183, 3062, 1658, 1612, 1509, 1484, 1389, 1300 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6): δ_H 8.28 (*s*, 1H, N–H), 7.61 (*d*, 1H, *J* = 7.08 Hz, Ar–H), 7.50–7.49 (m, 2H, Ar–H), 7.40–7.33 (m, 3H, Ar–H), 7.24 (*t*, 1H, *J* = 6.90 Hz, Ar–H), 7.10 (*s*, 1H, N–H), 6.75 (*d*, 1H, *J* = 8.04 Hz, Ar–H), 6.67 (*t*, 1H, *J* = 7.14 Hz, Ar–H), 5.75 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.5, 147.8, 141.6, 133.3, 128.6, 128.4, 128.3, 127.8, 127.3, 126.8, 117.1, 114.9, 114.4, 66.5; *m*/*z* (ESI), 225 $[M+H]^+$.

2-(4-Nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4b**) was isolated as bright-yellow crystals; m.p.: > 300 °C; IR (KBr): 3359, 3285, 3176, 1648, 1610, 1519, 1482, 1347, 1157, 1009, 859, 748 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6): δ_H 8.52 (*s*, 1H, N–H), 8.26 (*d*, 2H, *J* = 8.70 Hz, Ar–H), 7.75 (*d*, 2H, *J* = 8.64 Hz, Ar–H), 7.62 (*d*, 1H, *J* = 7.62 Hz, Ar–H), 7.32 (*s*, 1H, N–H), 7.27 (*t*, 1H, *J* = 6.69 Hz, Ar–H), 6.77 (*d*, 1H, *J* = 8.04 Hz, Ar–H), 6.69 (*t*, 1H, *J* = 7.35 Hz, Ar–H), 5.92 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.2, 149.3, 147.2, 133.5, 129.3, 128.0, 127.4, 125.9, 123.6, 123.5, 117.4, 114.9, 114.5, 65.3; *m*/*z* (ESI), 270 $[M+H]^+$.

2-(3-Nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4c**) was isolated as bright-yellow crystals; m.p.: 199–201 °C; IR (KBr): 3300, 3192, 3076, 1668, 1652, 1613, 1528, 1481, 1350, 1250, 1149, 746 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6): δ_H 8.53 (*s*, 1H, N–H), 8.37 (*s*, 1H, Ar–H), 8.21 (*d*, 1H, *J* = 7.14 Hz, Ar–H), 7.95 (*d*, 1H, *J* = 7.68 Hz, Ar–H), 7.70 (*t*, 1H, *J* = 7.95 Hz, Ar–H), 7.62 (*d*, 1H, *J* = 7.56 Hz, Ar–H), 7.34 (*s*, 1H, N–H), 7.28 (*t*, 1H, *J* = 7.17 Hz, Ar–H), 6.79 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.70 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.95 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.3, 147.7, 147.3, 144.3, 133.5, 133.3, 130.0, 127.4, 123.2, 121.5, 117.5, 114.9, 114.6, 65.2; *m*/*z* (ESI), 270 $[M+H]^+$.

2-(2-Nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4d**) was isolated as bright-yellow crystals; m.p.: 187–188 °C; IR (KBr): 3408, 3186, 3070, 2924, 1659, 1609, 1536, 1504, 1457, 1390, 1352, 1297, 1173, 1128, 1029 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6): δ_H 8.21 (*s*, 1H, N–H), 8.06 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 7.86–7.85 (m, 1H, Ar–H), 7.79 (*t*, 1H, *J* = 7.56 Hz, Ar–H), 7.66–7.61 (m, 2H, Ar–H), 7.26 (*t*, 1H, *J* = 7.02 Hz, Ar–H), 7.00 (*s*, 1H, N–H), 6.77 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.72 (*t*, 1H, *J* = 7.47 Hz, Ar–H), 6.33 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.3, 147.6, 147.1, 135.9, 133.9, 133.5, 129.9, 128.9, 127.3, 124.7, 117.6, 114.9, 114.5, 62.1; *m*/*z* (ESI), 270 $[M+H]^+$.

2-(*p*-Tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4e**) was isolated as white crystals; m.p.: 225–228 °C; IR (KBr): 3311, 3193, 3061, 1662, 1609, 1509, 1483, 1437, 1386, 1326, 1295, 1156, 1027 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6): δ_H 8.22 (*s*, 1H, N–H), 7.60 (*d*, 1H, *J* = 7.68 Hz, Ar–H), 7.37 (*d*, 2H, *J* = 7.74 Hz, Ar–H), 7.23 (*t*, 1H, *J* = 7.65 Hz, Ar–H), 7.18 (*d*, 2H, *J* = 7.68 Hz, Ar–H), 7.04 (*s*, 1H, N–H), 6.74 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.66 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.71 (*s*, 1H, CH), 2.29 (*s*,

3H, CH_3); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.6, 147.9, 138.6, 137.7, 133.2, 129.2, 128.8, 127.7, 127.3, 126.7, 117.0, 115.0, 114.4, 66.3, 20.7; *m/z* (ESI), 239 $[M+H]^+$.

2-(4-Methoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4f**) was isolated as white crystals; m.p.: 192–194 °C; IR (KBr): 3298, 3181, 3058, 1658, 1611, 1509, 1434, 1388, 1300, 1250, 1176, 1032 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.17 (*s*, 1H, N–H), 7.61 (*d*, 1H, *J* = 7.74 Hz, Ar–H), 7.42 (*d*, 2H, *J* = 8.64 Hz, Ar–H), 7.23 (*t*, 1H, *J* = 8.28 Hz, Ar–H), 6.99 (*s*, 1H, N–H), 6.94 (*d*, 2H, *J* = 8.58 Hz, Ar–H), 6.74 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.67 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.70 (*s*, 1H, CH), 3.74 (*s*, 3H, CH_3); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.7, 159.4, 148.0, 133.5, 133.2, 129.4, 128.2, 127.3, 117.0, 115.0, 114.4, 114.0, 113.6, 66.3, 55.1; *m/z* (ESI), 255 $[M+H]^+$.

2-(4-Chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4g**) was isolated as white crystals; m.p.: 200–203 °C; IR (KBr): 3307, 3187, 3063, 1659, 1609, 1509, 1484, 1433, 1386, 1294, 1156, 1092 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.32 (*s*, 1H, N–H), 7.61 (*d*, 1H, *J* = 7.62 Hz, Ar–H), 7.51–7.50 (m, 2H, Ar–H), 7.46–7.45 (m, 2H, Ar–H), 7.25 (*t*, 1H, *J* = 7.62 Hz, Ar–H), 7.13 (*s*, 1H, N–H), 6.75 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.68 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.77 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.4, 147.6, 140.7, 133.4, 132.9, 129.6, 128.7, 128.3, 127.3, 117.3, 114.9, 114.4, 65.7; *m/z* (ESI), 259 $[M+H]^+$.

2-(3-Chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4h**) was isolated as white crystals; m.p.: 188–190 °C; IR (KBr): 3291, 3195, 3065, 1651, 1612, 1512, 1481, 1436, 1386, 1297, 1159, 1097 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.38 (*s*, 1H, N–H), 7.61 (*d*, 1H, *J* = 7.74 Hz, Ar–H), 7.53 (*s*, 1H, Ar–H), 7.45–7.41 (m, 3H, Ar–H), 7.26 (*t*, 1H, *J* = 7.62 Hz, Ar–H), 7.20 (*s*, 1H, N–H), 6.76 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.69 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.78 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.4, 147.5, 144.4, 133.4, 132.9, 130.3, 128.2, 127.3, 126.7, 125.4, 117.3, 114.9, 114.4, 65.5; *m/z* (ESI), 259 $[M+H]^+$.

2-(2-Chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4i**) was isolated as white crystals; m.p.: 204–206 °C; IR (KBr): 3360, 3192, 3068, 1647, 1612, 1503, 1391, 1328, 1254, 1186, 1123, 1053, 853, 749 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.20 (*s*, 1H, N–H), 7.67–7.65 (m, 2H, Ar–H), 7.50–7.48 (m, 1H, Ar–H), 7.41–7.38 (m, 2H, Ar–H), 7.26 (*t*, 1H, *J* = 6.96 Hz, Ar–H), 7.01 (*s*, 1H, N–H), 6.76 (*d*, 1H, *J* = 8.04 Hz, Ar–H), 6.71 (*t*, 1H, *J* = 7.29 Hz, Ar–H), 6.14 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.6, 147.6, 137.9, 133.4, 131.8, 130.2, 129.5, 128.7, 127.4, 127.3, 117.4, 114.7, 114.5, 63.7; *m/z* (ESI), 259 $[M+H]^+$.

2-(4-Hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4j**) was isolated as white crystals; m.p.: 272–274 °C; IR (KBr): 3321, 3194, 1645, 1607, 1484, 1378, 1286, 1227, 1157, 869, 756 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 9.49 (*s*, 1H, OH), 8.08 (*s*, 1H, N–H), 7.61 (*d*, 1H, *J* = 6.96 Hz, Ar–H), 7.30 (*d*, 2H, *J* = 8.46 Hz, Ar–H), 7.23 (*t*, 1H, *J* = 6.93 Hz, Ar–H), 6.93 (*s*, 1H, N–H), 6.77–6.72 (m, 3H, Ar–H), 6.67 (*t*, 1H, *J* = 7.38 Hz, Ar–H), 5.65 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.7, 157.6, 148.1, 133.1, 131.6, 129.6, 128.2, 127.3, 117.0, 115.3, 114.9, 114.3, 66.6; *m/z* (ESI), 240 $[M+H]^+$.

2-(3-Hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4k**) was isolated as white crystals; m.p.: 202–205 °C; IR (KBr): 3324, 3190, 1644, 1611, 1485, 1466,

1361, 1283, 1227, 1157 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 9.49 (*s*, 1H, O–H), 8.23 (*s*, 1H, N–H), 7.61 (*d*, 1H, *J* = 7.68 Hz, Ar–H), 7.24 (*t*, 1H, *J* = 7.62 Hz, Ar–H), 7.17 (*t*, 1H, *J* = 7.77 Hz, Ar–H), 7.07 (*s*, 1H, N–H), 6.91–6.89 (m, 2H, Ar–H), 6.75–6.72 (m, 2H, Ar–H), 6.67 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.66 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.5, 157.3, 147.8, 143.2, 133.2, 129.3, 127.3, 117.4, 116.9, 115.3, 114.8, 114.3, 113.6, 66.4; *m*/*z* (ESI), 240 $[M+H]^+$.

2-(4-Fluorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4l**) was isolated as white crystals; m.p.: 202–205 °C; IR (KBr): 3300, 3182, 3066, 1658, 1610, 1508, 1483, 1438, 1388, 1296, 1233, 1156 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.28 (*s*, 1H, N–H), 7.62 (*d*, 1H, *J* = 7.44 Hz, Ar–H), 7.55–7.53 (m, 2H, Ar–H), 7.26–7.21 (m, 3H, Ar–H), 7.09 (*s*, 1H, N–H), 6.75 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.69 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.78–5.75 (m, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.5, 162.9, 161.3, 147.8, 137.8, 137.7, 133.3, 129.0, 127.3, 117.2, 115.1, 114.4, 65.9, 54.9; *m*/*z* (ESI), 243 $[M+H]^+$.

2-(4-(Dimethylamino)phenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4m**) was isolated as canary-yellow crystals; m.p.: 214–216 °C; IR (KBr): 3291, 3188, 1656, 1612, 1517, 1485, 1438, 1388, 1355, 1295, 1233, 1159 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.07 (*s*, 1H, N–H), 7.62 (*d*, 1H, *J* = 7.68 Hz, Ar–H), 7.31 (*d*, 2H, *J* = 8.58 Hz, Ar–H), 7.23 (*t*, 1H, *J* = 7.62 Hz, Ar–H), 6.92 (*s*, 1H, N–H), 6.75–6.72 (m, 3H, Ar–H), 6.67 (*t*, 1H, *J* = 7.44 Hz, CH), 2.89 (*s*, 6H, CH_3); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.8, 162.4, 152.3, 150.6, 148.2, 133.1, 128.9, 128.7, 127.7, 127.3, 116.9, 115.0, 114.4, 112,0, 111.2, 66.6; *m*/*z* (ESI), 268 $[M+H]^+$.

2-(2,4-Dichlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4n**) was isolated as white crystals; m.p.: 169–170 °C; IR (KBr): 3339, 3186, 3085, 2906, 1663, 1610, 1479, 1377, 1295, 1237, 1155, 1100, 1052, 1007 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.23 (*s*, 1H, N–H), 7.67–7.65 (m, 3H, Ar–H), 7.51–7.49 (m, 1H, Ar–H), 7.27 (*t*, 1H, *J* = 7.65 Hz, Ar–H), 7.02 (*s*, 1H, N–H), 6.76 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.73 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 6.12 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.5, 150.1, 147.5, 137.0, 133.9, 133.5, 132.9, 130.1, 128.9, 127.6, 127.4, 117.6, 114.6, 63.3; *m*/*z* (ESI), 293 $[M+H]^+$.

2-(Furan-2-yl)-2,3-dihydroquinazolin-4(1*H*)-one (**4o**) was isolated as yellow crystals; m.p.: 163–164 °C; IR (KBr): 3301, 3178, 3064, 2939, 1658, 1611, 1514, 1483, 1441, 1389, 1330, 1299, 1258, 1152, 1013 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.38 (*s*, 1H, N–H), 7.61–7.60 (m, 2H, Ar–H), 7.23 (*t*, 1H, *J* = 7.62 Hz, Ar–H), 7.20 (*s*, 1H, CH=), 6.75 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.67 (*t*, 1H, *J* = 7.47 Hz, Ar–H), 6.37 (*s*, 1H, N–H), 6.26 (*d*, 1H, *J* = 3.12 Hz, CH=), 5.74 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.2, 154.5, 147.1, 142.7, 133.2, 127.2, 117.2, 114.9, 114.4, 110.3, 107.1, 60.2; *m*/*z* (ESI), 214 $[M+H]^+$.

(*E*)-2-Styryl-2,3-dihydroquinazolin-4(1*H*)-one (**4p**) was isolated as white crystals; m.p.: 163–166 °C; IR (KBr): 3287, 3182, 3058, 2929, 1648, 1612, 1515, 1486, 1441, 1392, 1331, 1299, 1156, 1072, 1032 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.12 (*s*, 1H, N–H), 7.62 (*d*, 1H, *J* = 7.68 Hz, Ar–H), 7.45 (*d*, 2H, *J* = 7.56 Hz, Ar–H), 7.34 (*t*, 2H, *J* = 7.47 Hz, Ar–H), 7.28–7.24 (m, 2H, Ar–H), 6.88 (*s*, 1H, N–H), 6.76 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.70–6.66 (m, 2H, CH=), 6.37 (dd, 1H, J_1 = 15.84 Hz, J_2 = 6.78 Hz, Ar–H), 5.31 (*d*, 1H, *J* = 6.48 Hz, Ar–H); ^{13}C NMR

(150 MHz, DMSO-d_6): δ_C 163.3, 147.7, 135.7, 133.2, 131.6, 129.1, 128.7, 128.3, 128.1, 127.3, 126.6, 117.1, 114.8, 114.5, 65.8, 64.0; *m/z* (ESI), 251 $[M+H]^+$.

2-(3,4,5-Trimethoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4q**) was isolated as white crystals; m.p.: 189–190 °C; IR (KBr): 3322, 3212, 3066, 2937, 1649, 1608, 1495, 1460, 1420, 1357, 1326, 1299, 1126, 1040, 1004 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δH 8.19 (*s*, 1H, N–H), 7.62 (*d*, 1H, *J* = 7.68 Hz, Ar–H), 7.26 (*t*, 1H, *J* = 8.22 Hz, Ar–H), 7.03 (*s*, 1H, N–H), 6.85 (*s*, 2H, Ar–H), 6.77 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.69 (*t*, 1H, *J* = 7.47 Hz, Ar–H), 5.71 (*s*, 1H, CH), 3.77 (*s*, 6H, CH_3), 3.65 (*s*, 3H, CH_3); ^{13}C NMR (150 MHz, DMSO- d_6): δ_C 163.7, 163.6, 152.7, 148.0, 137.6, 136.6, 133.2, 127.3, 117.2, 115.0, 114.4, 104.4, 104.3, 66.8, 59.9, 55.9; m/z (ESI), 315 $[M+H]^+$.

2-(3,4-Dichlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**4r**) was isolated as white crystals; m.p.: 197–198 °C; IR (KBr): 3258, 3180, 3060, 2929, 1650, 1610, 1515, 1468, 1387, 1317, 1248, 1186, 1153, 1128, 1030 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6): δ_H 8.41 (*s*, 1H, N–H), 7.72 (*s*, 1H, Ar–H), 7.67 (*d*, 1H, *J* = 8.34 Hz, Ar–H), 7.61 (*d*, 1H, *J* = 7.62 Hz, Ar–H), 7.47 (*d*, 1H, *J* = 6.72 Hz, Ar–H), 7.27 (*d*, 1H, *J* = 7.65 Hz, Ar–H), 7.23 (*s*, 1H, N–H), 6.77 (*d*, 1H, *J* = 8.10 Hz, Ar–H), 6.70 (*t*, 1H, *J* = 7.44 Hz, Ar–H), 5.80 (*s*, 1H, CH); ^{13}C NMR (150 MHz, DMSO-d_6): δ_C 163.3, 147.3, 143.0, 133.5, 130.9, 130.8, 130.6, 128.9, 127.4, 127.0, 117.5, 114.9, 114.5, 65.0; *m/z* (ESI), 293 $[M+H]^+$.

Results and discussion

To evaluate the effect of different solvents on the condensation, the three-component one-pot reaction of isatoic anhydride, benzaldehyde, and ammonium acetate was chosen as model reaction in presence of $FeCl_3$/neutral Al_2O_3 (Table 1). We examined the reaction in different solvent systems (solvent free, MeOH, EtOH, *n*-propanol, *n*-butanol, CH_2Cl_2, THF, $CHCl_3$, H_2O, 1,4-dioxane, DMF, ethyl acetate, and *tert*-butanol) as depicted in Table 1. Among the tested conditions, solvent-free, MeOH, *n*-propanol, and *n*-butanol gave 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (**4a**) in poor yield with low turnover frequency (TOF) (Table 1, entries 1–4). Use of EtOH, CH_2Cl_2, THF, $CHCl_3$, H_2O, 1,4-dioxane, and DMF gave moderate yield with obviously increased TOF (Table 1, entries 5–11). When the experiment was carried out in ethyl acetate, the yield of **4a** was 81 % and the TOF was 8.77 h^{-1} (Table 1, entry 12). The best conversion was observed when the reaction was performed in *tert*-butanol, with **4a** being obtained in 92 % yield with TOF of 9.97 h^{-1} (Table 1, entry 13). Therefore, *tert*-butanol was selected as solvent for this reaction.

Moreover, we also found that the yield of the product **4a** was obviously affected by the amount of catalyst used in this reaction (Table 2). Without any catalyst, the product was obtained in 65 % yield (Table 2, entry 1). When the amount of catalyst was 20, 40, 60, 80, and 100 mg, formation of the product was observed in 85 %, 86 %, 90 %, 91 %, and 92 % yield (Table 2, entries 2–6), respectively. Using lower amounts of catalyst resulted in lower yield, while increasing the amount of catalyst above 100 mg did not improve the reaction yield (Table 2, entry 7). From these results, it can be concluded

that the optimum amount of the catalyst was 100 mg, giving **4a** in yield of 92 % (Table 2, entry 6).

To determine the optimum molar ratio of the substrates, the yields of the model reaction in *tert*-butanol using different molar ratios of isatoic anhydride, benzaldehyde, and ammonium acetate were obtained and compared (Table 3). As seen from Table 3, the best result was obtained using a ratio of 1:1.2:1.6 of isatoic anhydride, benzaldehyde, and ammonium acetate in presence of $FeCl_3$/neutral Al_2O_3 (Table 3, entry 8).

With the optimized conditions in hand, the reactions of different aromatic aldehydes with either electron-donating or electron-withdrawing groups were screened to explore the scope and generality of this protocol for synthesis of various 2,3-dihydroquinazolin-4(1*H*)-ones. The results are summarized in Table 4. As indicated in Table 4, aromatic aldehydes with electron-donating substituents performed well with isatoic anhydride and ammonium acetate in presence of $FeCl_3$/neutral Al_2O_3 in *tert*-butanol under reflux conditions, affording the desired products in excellent yield (Table 4, entries 5, 6, 13). However, aldehydes with strongly electron-withdrawing groups on aromatic ring gave the products in moderate yield under the similar reaction conditions (Table 4, entries 2–4). These observations indicate that the electronic effect and the position of the substituent group on the phenyl ring had some impact on this conversion.

We also examined the reaction of furfuraldehyde with isatoic anhydride and ammonium acetate, which gave the desired product 2-(furan-2-yl)-2,3-dihydroquinazolin-4(1*H*)-one (**4o**) in good yield (81 %) (Table 4, entry 15). 3,4,5-Trimethoxybenzaldehyde was also condensed with isatoic anhydride and ammonium acetate successfully; the corresponding 2-3,4,5-trimethoxyphenyl-2,3-dihydroquinazolin-4(1*H*)-one (**4q**) was obtained in good yield within 4 h (Table 4, entry 17).

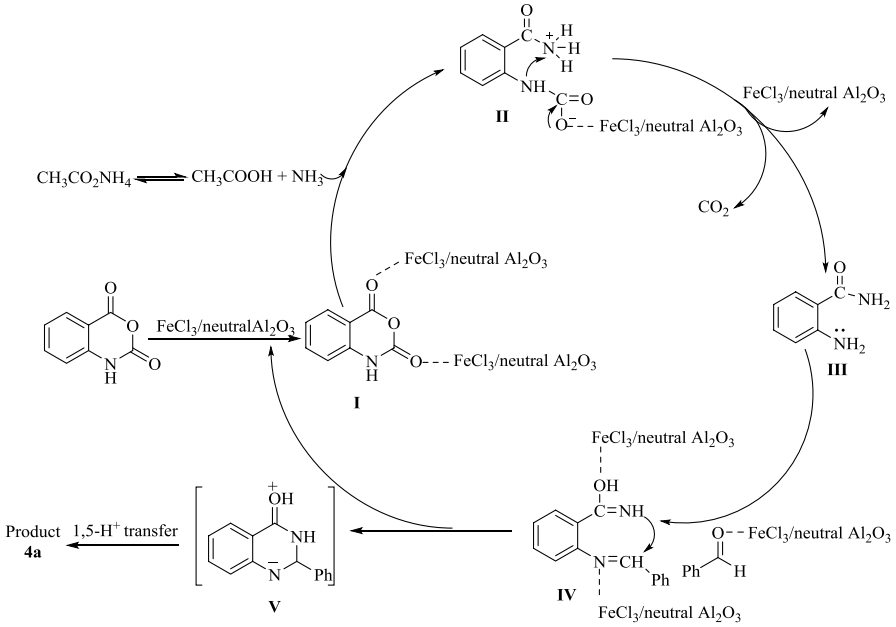
No product was obtained when 2,4-dinitrobenzaldehyde and 2,3,4-trimethoxybenzaldehyde (Table 4, entries 19, 20) were used in this experiment. The reactions of aliphatic aldehydes could not afford the corresponding products even when prolonging the reaction time (Table 4, entries 21, 22). It seems that this protocol has limitations with respect to these substrates.

Recovery and reusability are the most important features of supported catalysts. The recyclability of the catalyst was investigated using a model reaction (Table 5). After reaction completion, the catalyst was separated by simple filtration and washed with ethyl acetate, air-dried, and reused directly in the same reaction. The experimental results revealed that the catalyst can be reused, albeit accompanied by appreciable loss of activity.

Based on the observations in the progress of the experiments and other mechanisms reported in literature [29, 30, 33, 35, 36], a plausible mechanism for the synthesis of **4a** is proposed in Scheme 2. Firstly, isatoic anhydride is activated by $FeCl_3$/neutral Al_2O_3 to produce reactive intermediate **I**, followed by attack of NH_3

Table 5 Synthesis of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one with recovered $FeCl_3$/neutral Al_2O_3

Number of cycles	Fresh	Recycle I	Recycle II	Recycle III
Isolated yield (%)	92	87	82	78



Scheme 2 Proposed mechanism for synthesis of 2-phenyl-2,3-didihydroquinazolin-4(1*H*)-one

on the carbonyl to form intermediate **II**, which in turn is converted to 2-amino-*N*-substituted-amide (**III**) by decarboxylation and proton transfer reaction. Subsequently, reaction of benzaldehyde activated by $FeCl_3$/neutral Al_2O_3 with **III** gives imine intermediate **IV**. The amide part of **IV** could be converted into tautomer in the presence of $FeCl_3$/neutral Al_2O_3. Meanwhile, intermediate **V** is obtained by intermolecular nucleophilic attack of the amide nitrogen on imine carbon. Finally, 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (**4a**) can form by 1,5-proton transfer of **V**.

Conclusions

We developed an ecofriendly method to synthesize 2,3-dihydroquinazolin-4(1*H*)-one derivatives by three-component reaction of isatoic anhydride, aromatic aldehyde, and ammonium acetate using $FeCl_3$/neutral Al_2O_3 as catalyst in *tert*-butanol under reflux condition. The advantages of this methodology are milder conditions, shorter reaction time, good yield, and environmentally benign catalyst.

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